Ecofriendly Dyeing and Antibacterial Finishing of Soyabean Protein Fabric Using Waste Flowers from Temples

M. D. Teli*, Javed Sheikh, Maruti Kamble

Department of Fibres and Textile Processing Technology,

Institute of Chemical Technology

Matunga (E), Mumbai-400019, India

mdt9pub@gmail.com; javed_uict@yahoo.co.in

Abstract

Soyabean Protein Fibre (SPF) is considered to be important regenerated protein fibre for various applications in textiles because of its unique properties. However the lack of antibacterial properties of such protein containing polymers is held as a severe limitation for its applications in hygienic textiles and the need to make it antibacterial is quite intense. A lot of marigold (which is antibacterial), used in Idol worship forms a temple waste and there is tremendous potential to use this waste as a good source of natural dye. In the current study, the tannin mordants were extracted from tamarind seed coats, amla (*Indian gooseberry*) and harda (*Myrobalan fruits*) and their application in natural dyeing using temple waste marigold as a dye was carried out. Marigold dyeing using most commonly found alum mordant was also carried out for comparison of the purpose. The dyed SPF fabrics were then evaluated for colour values, fastness properties, antibacterial activities as well as durability of the same. The results clearly indicated the advantages of using such mordants both in case of achieving antibacterial functionality as well as eco-friendliness.

Keywords

SPF; Naturaldyeing; Naturalmordants; Antibacterial properties.

Introduction

Textiles, especially those made of natural fibers, are an excellent medium for the growth of microorganisms when the basic requirements for their growth such as nutrients, moisture, oxygen, and appropriate temperature are present. The large surface area and ability to retain moisture of textiles also assist the growth of microorganisms on the fabric (Su et al., 2011) which intern results in inflicting a range of unwanted effects not only on the textile itself but also on the wearer. These effects include the generation of unpleasant odor, stains, discoloration in the fabric, a reduction in the tensile strength of the fabric and an increased likelihood of contamination(Gao and Cranston, 2008). In the last few decades, with the increase in new antimicrobial fibre technologies and the growing awareness about cleaner surroundings and healthy lifestyle, a range of textile products based on synthetic antimicrobial agents such as triclosan, metal and their salts, organometallics, phenols and quaternary ammonium compounds, have been developed and quite a few were also available commercially (Joshi et al, 2009). Even though the excellent antimicrobials are available, their effect on ecology is always a question. Under this backdrop some of the natural dyes reported to possess antimicrobial properties attract the attention of the researchers in this field.

On the other hand the development of synthetic dyes taking place at the beginning of the twentieth century has come a long way and led to a more complete level of quality and more reproducible techniques of application. Due to wide applications on variety of fibres, and the economies of scale, considerable reduction in the dyestuff costs per kg of dyed goods has been achieved making their application economical. However during the last two decades, the use of synthetic dyes is gradually receding due to an increased environmental awareness and harmful effects caused by either toxic degraded products or their non-biodegradable nature. In addition to above, some serious health hazards like allergenicity and carcinogenecity are associated with the synthetic dyes. As a result, recently a ban has been imposed all over the world including European Economic Community (EEC), Germany, USA and India on the use of some synthetic dyes (e.g. azodyes) which finally triggered active research and development to revive world heritage and traditional wisdom of employing safer

natural dyes. Consumers nowadays are becoming more and more concerned about environmental issues and hence the demand for natural product incorporating natural ingredient soars. Thus natural dyes are gaining increasing importance as they are obtained from renewable resources and free from health hazards and some of them sometimes act as a health care products too (Bechtold et al, 2003; Kumary and Sinha, 2004; Prabhu et al, 2011).

However, natural dyes in general, with few expectations are non-substantive and hence it must be used in conjunction with mordants. Mordant is a chemical, which can fix itself on the fibre and also combines with the dyestuff. The challenge before the natural dyers in application of natural colour is necessity to use metallic mordants which themselves are pollutant and harmful. Due to the environmental hazard caused by metallic mordant while dyeing of textile fabric, manufacturers have to find out safe natural mordant for application of natural dyes.

Although a lot of work has been done on application of natural dyes on textile fabric, in most of the cases metal mordants used are environmentally objectionable. Tannin is an astringent vegetable product found in a wide variety of plant parts such as bark, wood, fruit, fruit pods, leaves, roots and plant galls. Tannins are defined as naturally occurring water soluble polyphenolic compounds of high molecular weight (about 500–3000) containing phenolic hydroxyl groups to enable them to form effective crosslinks between proteins and other macromolecules (Ramakrishnan et al, 2006). They are primarily used in the preservation of leather (Swarna et al, 2009), glues, stains and mordants. Application of tannin based natural mordants in natural dyeing was reported earlier from our laboratory (Prabhu et al, 2011; Teli and Prabhu, in press;Teli et al, 2012) .

There is common practice in India of throwing the temple flowers once used in Idol worship into river, which contributes to the water pollution. The waste disposal of such flowers is itself an issue and hence exploring the potential of using this flower waste from temples for dyeing of textile has been undertaken. Dyeing of textiles with marigold flower as a dye as such has been reported earlier (Teli et al, 2012; Vankar et al, 2009; Jothi 2008; Ujjin and Suesat, 2012; Saha and Datta, 2008; Sarkar et al, 2006; Sarkar et al, 2005; Samanta, 2008). Tannins are astringent and antimicrobial in nature. Such properties are also displayed by marigold extract. The application of natural dyes using natural mordants hence can act in dual way of natural coloration and antibacterial finishing for textile materials. Even though tannin containing plants are available in plenty, the application of such sources for extracting mordants to be used in natural dyeing has been explored to a very limited extent. In the current work, the simultaneous dyeing and antibacterial finishing of Soyabean Protein Fibre (SPF) was carried out using natural mordants extracted from harda (*myrobalan)*, tamarind seed coat (TSC) and amla. The colour values were evaluated and compared with those obtained using alum mordant. The antibacterial efficacy of the dyed material and the durability of the antibacterial activity were studied.

Materials

SPF yarn (30 count) was knitted to make fabric (single jersey) which was hot washed, bleached and used for dyeing. All chemicals used were of laboratory grade. Marigold was obtained from ISCON temple, Mumbai.

Methods

Extraction of Mordant

The 1% stock solution of alum was made by dissolving 10 gm of mordant powder in 1000 ml water. In case of natural mordants, the 1% stock solution was made by boiling 10 gm of mordant powder in 1000 ml water for 1 h. The extract was filtered and made to 1000ml which was used for mordanting of the fabric.

Extraction of Dye

The 1% stock solution of the marigold dye was prepared by boiling 10 g of dry marigold flower in 1000 ml water for 1 h. The extract was filtered and made to 1000 ml and used for dyeing.

Mordanting and Dyeing of SPF

The mordanting of SPF fabric was carried out in rota dyer (Rota Dyer machine, Rossari® Labtech, Mumbai) keeping the liquor to material ratio of 30:1. The fabrics were introduced into the mordant solution at room temperature and slowly the temperature was raised to 95 ^{0}C. The mordanting was continued at this temperature for 30 min. After mordanting, the fabric was squeezed and dyed using marigold flowers extract as a dye. The mordanted fabrics were introduced into dyebath and dyeing was continued at 900C for 1h. After dyeing, the fabrics were squeezed and washed with cold water.

Colour value by Reflectance Method

The dyed samples were evaluated for the depth of colour by reflectance method using 10 degree observer. The absorbance of the dyed samples was measured on Spectraflash SF 300 (Datacolor International, U.S.A.) equipped with reflectance accessories. The K/S values were determined using expression;

$$K/S = \frac{(1-R)^2}{2R}$$

where, R is the reflectance at complete opacity, K is the Absorption coefficient and S is the Scattering coefficient.

Dyed fabrics were simultaneously evaluated in terms of CIELAB colour space (L*, a* and b*) values using the Spectraflash SF300. In general, the higher K/S value represents higher depth of colour on the fabric. L* corresponds to the brightness (100= white, 0= black), a* to the red–green coordinate (+ve= red, -ve =green) and b* to the yellow–blue coordinate (+ve =yellow, -ve =blue). As a whole, a combination of all these enables one to understand the tonal variations.

Washing Fastness

Evaluation of colour fastness to washing was carried out using ISO II methods (Trotmann, 1984). A solution containing 5 g/L soap solution was used as the washing liquor. The samples were treated for 45 min at 50^0C using liquor to material ratio of 50:1 in rota machine. After rinsing and drying, the change in colour of the sample and staining on the undyed fabric samples were evaluated on the respective standard scales (rating 1–5, where 1 – poor, 2 – fair, 3 – good, 4 – very good and 5 – excellent).

Rubbing Fastness

Evaluation of colour fastness to rubbing (dry and wet) was carried out using "crock-meter" with 10 strokes of rubbing.

Light Fastness

Dyed fabric was tested for colour fastness to light according to ISO 105/B02 (ISO technical manual, 2006). The light fastness was estimated using artificial illumination with Xenon which is a light source, Q-Sun Xenon Testing Chamber at black standard temperature 65 ^{0}C with relative humidity of the air in the testing chamber as 40% and daylight filter, wavelength, k= 420 nm. The samples were compared with the standard scale of blue wool reading (ratings, 1–8, where 1 – poor, 2 – fair, 3 –moderate, 4 – good, 5 – better, 6 – very good, 7 – best and 8 –excellent).

Determination of Antimicrobial Activities of Dyed Fabrics

The antibacterial activity of the dyed fabrics was estimated by AATCC Test Method 100-2004 (AATCC technical manual, 2007). The reduction in number of bacterial colonies formed with respect to the untreated control sample was estimated by using following equation,

$$R = \frac{100\,(B-A)}{B}$$

where,

R = % reduction in bacterial count;

A = the number of bacterial colonies recovered from the inoculated treated test specimen swatches in the jar incubated for 24 hr contact period;

B = the number of bacterial colonies recovered from the inoculated untreated control test specimen swatches in the jar immediately after inoculation (at "0" contact time).

Durability of antimicrobial activity

The durability to laundering was measured using washing conditions as per ISO 105-CO6-1M [19].

Results and Discussion

Optimization of Mordant and Dye Concentrations

The dyeing of SPF fabric employing most commonly used metal mordant alum and natural mordants like harda, amla and tamarind seed coat (TSC) was attempted and results are summarized in Tables 1-5.

Initial attempt of the study was to find the contribution of mordant and dye towards colour values of the dyed SPF fabrics. Hence SPF fabrics were initially in one case just mordanted but not dyed and in second case dyed without pre-mordanting. The results in Table 1 showed the increase in K/S values with increasing concentration of mordants. It must be noted that SPF fabrics used for dyeing were initially slightly yellowish and hence showing some colour values without any mordanting or dyeing (refer Table 1). Alum showed least effect on colour values with increasing concentration from 5% to 20%. However the tannin mordants showed marked increase in K/S with increasing mordant concentration. Among the

tannin mordants, amla tannin mordant showed the highest increase in K/S followed by harda and tamarind seed coat. In general, the increasing concentration of tannin mordants showed the increase in redness of shades. The effect of marigold dye concentration on colour values was also studied and results are summarized in Table 2.

TABLE 1 EFFECT OF MORDANT CONCENTRATION ON COLOUR VALUES OF MORDANTED SPF SAMPLES

Mordant	Mordant conc. (%)	K/S	L*	a*	b*
-	-	0.7348	81.279	2.333	22.172
Alum	5	1.2446	74.291	1.439	22.537
	10	1.3994	73.128	0.754	20.975
	15	1.4118	74.694	1.544	23.659
	20	1.4693	73.469	4.095	23.251
Harda	5	2.9399	60.723	2.096	16.725
	10	2.9865	63.185	2.542	19.774
	15	3.7779	62.779	2.533	19.132
	20	3.8037	63.743	2.941	20.289
TSC	5	1.8239	62.611	5.474	20.257
	10	1.9895	62.747	6.305	20.272
	15	2.2879	60.853	6.411	18.329
	20	2.3663	60.151	7.269	17.606
Amla	5	2.8717	58.163	3.883	17.081
	10	3.1386	59.4	3.923	18.503
	15	3.8041	58.903	4.216	18.3
	20	4.2542	58.595	4.175	17.752

TABLE 2 EFFECT OF DYE CONCENTRATION ON COLOUR VALUES OF ONLY-DYED SPF SAMPLES

Dye	Dye Conc. (%)	K/S	L*	a*	b*
Marigold	5	2.8686	66.085	1.844	22.632
	10	3.7455	65.77	1.433	21.748
	15	4.0097	62.028	3.439	23.052
	20	4.2397	60.888	3.566	22.208

TABLE 3 EFFECT OF VARYING CONCENTRATION OF ALUM AND MARIGOLD ON COLOUR VALUES OF DYED SPF FABRIC

Alum (%)	Marigold (%)	K/S	L*	a*	b*
5	5	5.5228	66.503	4.355	40.756
5	10	10.0529	66.442	2.726	38.444
5	15	11.8729	67.955	2.494	40.142
5	20	13.4266	60.742	4.682	43.456
10	5	6.4807	54.323	7.55	39.602
10	10	10.2255	57.923	6.135	43.042
10	15	10.9399	60.551	5.577	43.867
10	20	14.2789	68.874	2.044	39.643
15	5	6.9268	66.975	4.445	40.6
15	10	10.577	58.389	4.69	40.499
15	15	14.641	61.319	4.491	43.981
15	20	17.8174	58.786	4.622	43.592
20	5	7.3248	53.109	3.367	33.784
20	10	12.2046	57.255	5.261	41.354
20	15	13.2829	58.661	5.324	43.309
20	20	17.9598	58.955	4.931	43.635

TABLE 4 EFFECT OF VARYING CONCENTRATION OF HARDA AND MARIGOLD ON COLOUR VALUES OF DYED SPF FABRIC

Harda (%)	Marigold (%)	K/S	L*	a*	b*
5	5	3.8059	59.989	3.815	22.823
5	10	3.9802	60.794	3.348	22.195
5	15	4.6245	66.897	1.721	22.338
5	20	5.2931	68.401	1.277	22.828
10	5	4.1157	58.313	2.647	18.26
10	10	5.4387	58.423	3.833	24.827
10	15	5.5844	56.737	3.789	23.531
10	20	6.1405	57.532	3.771	23.359
15	5	5.0177	56.43	4.14	23.707
15	10	5.7317	65.096	3.067	24.264
15	15	5.7487	56.074	3.747	23.257
15	20	6.5905	59.177	3.575	24.976
20	5	5.1231	54.98	4.213	22.963
20	10	5.6716	58.058	4.134	24.591
20	15	5.8756	59.135	3.528	24.259
20	20	7.5429	64.339	2.523	23.862

The K/S values showed increase with increasing dye concentration from 5% to 20%. The SPF showed good dyeability towards marigold dye in absence of mordants, which might be due to the presence of amino groups in protein fibres like SPF which imparts substantivity towards the dye molecules. The rise in dye concentration also resulted in increase in redness of shades.

The results of dyeing of marigold using different mordants are summarized in Tables 3-6.

The K/S values were found to be improved with increasing alum concentration till 20%; but the relative increase in K/S values from mordant concentration of 15 % to that of 20% was comparatively lower. Hence 15% mordant concentration was taken as an optimum concentration. For a constant alum concentration, K/S values were found to be increased with marigold dye concentration from 5% to 20%. The colour value thus obtained, in the case of natural dyes is a combined contribution of the effect of mordant and the dye. Hence the K/S was improved with mordant and dye concentration initially till the equilibrium was reached. The increase in concentrations of either mordant or dye beyond optimum concentrations did not significantly contribute to the improvement of the depth of dyeing which is reflected in K/S values.

TABLE 5 EFFECT OF VARYING CONCENTRATION OF TAMARIND SEED COAT AND MARIGOLD ON COLOUR VALUES OF DYED SPF FABRIC

TSC (%)	Marigold (%)	K/S	L*	a*	b*
5	5	3.1716	57.521	5.263	19.836
5	10	3.3931	56.043	6.303	18.757
5	15	3.7691	59.234	4.967	20.474
5	20	5.2415	52.322	3.279	13.519
10	5	3.5341	55.684	6.42	18.19
10	10	3.7573	57.479	6.211	19.698
10	15	3.9831	55.447	7.731	19.901
10	20	5.2722	57.642	6.808	20.923
15	5	3.6433	53.455	7.366	18.365
15	10	3.879	55.522	7.19	19.941
15	15	4.7567	60.272	5.984	21.011
15	20	5.3063	53.636	7.732	21.596
20	5	5.2149	59.647	7.065	22.212
20	10	5.2149	55.183	7.825	22.603
20	15	5.3063	58.16	7.06	21.309
20	20	6.0307	62.653	5.363	22.324

TABLE 6 EFFECT OF VARYING CONCENTRATION OF AMLA AND MARIGOLD ON COLOUR VALUES OF DYED SPF FABRIC

Amla (%)	Marigold (%)	K/S	L*	a*	b*
5	5	2.9932	59.741	3.697	18.686
5	10	4.0619	59.83	3.858	18.714
5	15	4.5883	64.118	3.319	21.258
5	20	4.7118	62.274	3.176	20.012
10	5	4.0555	61.247	3.666	19.474
10	10	4.2796	61.026	3.315	19.092
10	15	5.5623	54.517	4.456	21.355
10	20	5.9055	53.447	4.116	20.306
15	5	4.2504	50.999	5.307	19.553
15	10	4.5406	53.81	4.309	20.984
15	15	5.6058	51.26	4.162	18.839
15	20	6.3601	53.206	4.362	20.44
20	5	4.5298	50.599	4.829	19.08
20	10	5.9158	52.435	4.814	20.199
20	15	6.401	54.839	4.348	21.749
20	20	6.5611	53.625	4.554	21.15

Results in Table 4 clearly indicate the increase in K/S values with increasing harda concentrations from 5 to 20% which was more or less similar in case of all the natural mordants. However the colour values got almost leveled-off for 15% mordant concentration which was also the case when SPF fabrics were mordanted (Table 1). For a constant mordant concentration, K/S values increased with increasing dye concentration. The higher K/S values obtained for the pre-mordanted and dyed fabric as compared to that of the fabric sample which was not mordanted before dyeing clearly showed the role of natural tannins acting as a mordant in natural dyeing. Even though these natural mordants showed good colour values for marigold dyeing, they were found to be lower than those obtained using alum as a mordant. The tonal variations in shades of dyed SPF fabrics, as indicated by a* and b* values, were found to be varied with mordant and dye combinations (refer Tables 3-6); however the different shades were obtained using different mordants and dye combinations enabling one to have wider choice of hues from the gamut of different shades obtained in case of natural dyeing.

The results in Table 7 clearly indicate the fastness properties of the dyeings obtained using various mordants. The only dyed samples showed much inferior fastness properties as compared to those pre-mordanted and dyed. In other words, it indicates that the mordants play an important role in holding the dye on the fabric.

TABLE 7 EFFECT OF MORDANT TYPE (20%) AND MARIGOLD (20%) ON FASTNESS PROPERTIES

Mordant	Mordant conc. (%)	Marigold Conc. (%)	Fastness Properties		
			Washing	*Rubbing*	*Light*
Alum	-	20	2	3-4	2
	15	20	4-5	4-5	5
	20	20	4-5	4-5	5
Harda	15	20	4	4	5
	20	20	4	4-5	5
TSC	15	20	4-5	4-5	5
	20	20	4	4-5	5
Amla	15	20	4	4-5	5
	20	20	4	4-5	5

The washing fastnesses obtained varied in the range of very good to excellent grade. The rubbing fastness was also found to be of the grade good to excellent. Light fastness was found to be quite satisfactory.

Antibacterial Activity of Marigold Dyed SPF Fabric

The quantitative antibacterial assessment was made using AATCC-100(2004) test method and the results are presented in Tables 8-9.

The antibacterial activities of samples only mordanted and the samples only dyed as well as that of samples both mordnted and dyed are given in Table 8. The only dyed sample showed least extent of antibacterial

activity among the three categories of the samples. The antibacterial property is higher in the samples only mordanted that that in the samples only dyed. Whereas, mordanted and dyed samples showed the highest antibacterial property. All the three natural mordants gave more or less similar extent of overall antibacterial activity on dyeing with marigold.

Results in Table 9 indicate that the washing durability of such antibacterial property, irrespective of the mordant used was up to 20 cycles which is based on the assumption that minimum 70% reduction in bacterial count is an acceptable limit. It is also to be noted that among the three natural mordants tried, washing durability of the antibacterial property was slightly better in case of TSC and Amala, as compared to Harda which is generally used in natural dyeing as a source of tannin and this was clear when dyed samples after 20 washes were compared.

TABLE 8 EFFECT OF MORDANT TYPE (20%) AND MARIGOLD (20%) ON ANTIBACTERIAL PROPERTIES

Mordant	**Mordant conc. (%)**	**Marigold conc. (%)**	**Bacterial Reduction (%)**	
			S.aureus	***E. coli***
Alum	20	-	62.25	60.80
	-	20	45.05	44.75
	20	20	90.30	86.75
Harda	20	-	63.98	62.58
	20	20	90.20	87.25
TSC	20	-	62.20	61.75
	20	20	89.75	87.10
Amla	20	-	60.80	60.10
	20	20	88.65	86.25

TABLE 9 DURABILITY OF ANTIBACTERIAL PROPERTIES OF MARIGOLD DYED SPF

Mordant	**Mordant conc. (%)**	**Marigold conc. (%)**	**No. of Washes**	**Bacterial Reduction (%)**	
				S.aureus	***E. coli***
Alum	20	20	0	90.3	86.8
			5	84.2	83.2
			10	81.1	80.3
			20	73.3	71.5
			30	65.3	64.9
Harda	20	20	0	90.2	87.25
			5	81.20	81.20
			10	75.30	73.75
			20	69.75	68.25
			30	62.50	61.60
TSC	20	20	0	89.75	87.10
			5	86.00	84.10
			10	79.25	78.40
			20	74.25	73.05
			30	65.55	64.80
Amla	20	20	0	88.65	86.25
			5	83.15	81.50
			10	78.40	76.15
			20	73.70	71.30
			30	64.30	62.55

Conclusions

Ecofriendly dyeing and antibacterial finishing of SPF fabric was successfully carried out using natural mordants and marigold dye. The dyed product displayed good colour strength, although it was distinctly lower than that in those obtained using metal mordant like alum. The fastness properties were however, comparable for both types of mordants. The natural mordanted-dyed samples displayed broad spectrum and durable antibacterial activity. The marigold flower waste thus can be productively used not only for dyeing of SPF fabric, but also to impart antibacterial property. The replacement of metal mordant by the newly found the natural mordents TSC and Amla which individually contributes towards antibacterial property, further showed enhanced level in such property upon dyeing with marigold. The concept of natural dyeing of SPF fabric using natural mordant with an aim to produce gamut of shades and at the same time the impartment of antibacterial property seems to be indeed promising one.

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M.D.Teli is serving as a senior most Professor in Textile Chemistry at Institute of Chemical Technology (Elite statuts deemed university)., Mumbai and has a teaching and research experience of 33 years. He has published more than 300 papers in various national and international journals and guided 19 Ph.D. students and 77 M.Tech. students.